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(54) PROCESS FOR THE FINISHING OF TEXTILE MATERIALS

(71) We, FARBENFABRIKEN BAYER AKTIENGESELLSCHAFT, a body corporate organised under the laws of Germany, of Leverkusen, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for the finishing of textile materials; more particularly it concerns a process wherein the textile materials are treated with an aqueous liquor which contains a silica sol and an socyanate group-carrying reaction product from a compound of molecular weight of from 500 to 6000, containing at least two hydroxyl groups per molecule, with a polyisocyanate or bisulphite addition product thereof; Preferably the textile materials are treated according to the exhaust process.

The silica sols to be used in the process according to the invention may be any of the commercial silica sols; as is well known these generally contain 10-30% by weight of silica, generally of a particle size of between 10 and 50 mμ. The amount in which the silica sols are added to the aqueous liquors may vary within wide limits; in general, an addition of 0.3-5 g per litre of treating liquor has proved to be advantageous.

The isocyanate group-containing reaction products used in the aqueous liquors are obtained in known manner by reacting the compounds of molecular weight 500 to 6000 which contain at least two hydroxyl groups, with a stoichiometric excess, calculated on the hydroxyl content, of polyisocyanates or their bisulphite addition products at elevated temperatures.

Examples of compounds of molecular weight 500 to 6000 which contain at least two hydroxyl groups are polyalkylene ether glycols, such as polyethylene, polypropylene, polybutylene or polyhexylene glycol; polyalkyl ether polyols, e.g. polyalkyl ether triols, such as the addition copolymer products of ethylene oxide or propylene oxide and trimethylol propane; furthermore polyesters, as are obtained, for example, from aliphatic dicarboxylic acids, such as succinic acid, adipic acid, sebacic acid or maleic acid, and polyhydric alcohols, such as ethylene glycol, diethylene glycol, propylene glycol, butanediol and neopentyl glycol.

Examples of polyisocyanates are aliphatic diisocyanates, such as tetramethylene diisocyanate, hexamethylene diisocyanate, 1,4-cyclohexane diisocyanate, 4,4′ - dicyclohexyl - methane diisocyanate, and 1 methylcyclohexane 2,4 or 2,6-diisocyanate; aromatic diisocyanates, such as *p*-phenylene diisocyanate and 2,4- or 2,6-toluylene diisocyanate; as well as triisocyanates, such as the reaction product of the formula

$$OCN - (CH_2)_6 - N - [CONH - (CH_2)_6 - NCO]_2$$

which can be obtained from 3 moles of hexamethylene diisocyanate and 1 mole of water.

The content of isocyanate group-containing reaction products in the aqueous liquors may vary within wide limits; in general, amounts of 0.2 to 20 g, preferably 0.5 to 5 g, per litre of treating liquor have proved satisfactory.

In order to stabilise the liquor which contains the isocyanate group-carrying reaction product, it is expedient to add an emulsion stabiliser to the liquor. The emulsion stabiliser is, for example, an anionic surfactant, such as fatty alcohol sulphates or paraffin sulphonates, and, in particular, non-ionic compounds, such as the polymers or copolymers prepared from vinyl or divinyl monomers.

The polymers and copolymers used as emulsion stabilisers in the aqueous liquors

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may be based on the following vinyl or divinyl monomers, for example: ethylene, propylene, vinyl chloride, vinyl acetate, vinyl, ethers, such as vinyl ethyl ether; furthermore, styrene or divinyl-benzene, butadiene, isoprene or chloroprene; and α,β - unsaturated carboxylic acids, such as acrylic acid and methacrylic acid as well as their nitriles, esters and amides. Polymers which contain groups capable of reacting with isocyanates have proved particularly advantageous; for example, the polymers or copolymers prepared from acrylic acid, methacrylic acid, their hydroxyalkyl esters or amides; as well as the copolymers which are obtained when N - methylol - acrylamide, N - methylol - methacrylamide, or their derivatives prepared by the reaction with alcohols containing at least one further functional group, are copolymerised with other olefinic-unsaturated compounds, for example, according to the process of United States Patent Specification No. 3,243,399 (= British Patent Specification No. 1,002,451).

The amounts in which the emulsion stabilisers are added to the aqueous liquors may vary within wide limits; however, it has proved particularly advantageous, for example, to add fatty alcohol sulphates or paraffin sulphonates in an amount of 0.01 to 2 g, preferably 0.05 to 0.2 g, per litre of liquor, and the polymers or copolymers in an amount of 0.1 to 20 g, preferably 1 to 3 g, per litre of liquor.

In order to reduce the period of time during which the polymers and isocyanate group-containing reaction products draw on the textile materials, it is frequently advisable to add electrolytes to the treating baths, for example, alkali metal and ammonium salts of inorganic acids, e.g. sodium sulphate, ammonium phosphate, or alkali metal and ammonium salts of organic acids, e.g. sodium acetate and ammonium acetate. The amounts in which the electrolytes are added to the liquors may vary within wide limits; in general, amounts of 2 to 10 g per litre of liquor have proved satisfactory.

The treatment of the textile materials according to the process of the invention can be carried out, for example, by agitating the textile materials at room temperature in a liquor ratio of 1 : 6 to 1 : 50 in the aqueous liquor which contains an isocyanate group-carrying reaction products as hereinbefore described and optionally contains an emulsion stabiliser and the pH value of which amounts to about 4 to 6, for a short time, about 1 to 20 minutes; then adding the silica sol; again allowing the liquor to act for a short time, about 5 to 30 minutes; then, optionally after the addition of an electrolyte and another brief action of the liquor for about 5 to 30 minutes, centrifuging or squeezing; and subsequently drying.

The textile materials treated in this way, such as yarns, textured yarns, fabrics, knitted fabrics or finished articles of fabrics and knitted fabrics, may subsequently be subjected to other finishing processes, e.g. dyeing, optionally after an ageing treatment with H_2O_2 or after a short storage for about 1 to 2 days.

With the aid of the process according to the invention it is possible to impart an excellent finish to textile materials of natural polyamides, such as wool and silk, case in fibres, or synthetic fibres, such as polyamide, polyurethane, polyester, polycarbonate, polyacrylonitrile, polypropylene fibres. The treated textile materials are characterised by excellent properties in use, such as resistance to abrasion, creasing, and pilling and by insensitivity to soiling. In textile materials of wool, moreover, an excellent felt-free finish is achieved, which is fast to washing. The feel of the textile materials can also be advantageously affected by the process according to the invention.

The parts given in the Examples are parts by weight.

Example 1

A pre-washed worsted fabric of pure wool is treated in a washing machine in a liquor ratio of 1 : 5 at room temperature for about 10 minutes with an aqueous liquor containing, per litre,

60 g of a mixture
prepared by stirring into
below
12 g of the 50% stock emulsion described first
3 g of the 40% aqueous copolymer dispersion described below, and then
45 ml of water.

Before adding the mixture, the pH value of the liquor had been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

2 g of 30% silica sol,
diluted with 18 ml of water and acidified to pH 5-6 with acetic acid,

and the treatment is continued for another 30-40 minutes. Subsequently there is added to the liquor, per litre,

6 g of sodium acetate,
dissolved in 40 ml of water,

and the treatment is again continued for 10-15 minutes. The worsted fabric is then centrifuged and dried on a stenter at 100°C. By the treatment there is obtained an excellent felt-free finish which is fast to washing and a very good creasing resistance of the worsted fabric.

The 50% stock emulsion used above was obtained in the following way:

1000 parts of an 80% solution of the isocyanate group-containing reaction product described below, in ethyl acetate were slowly

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poured, while vigorously stirring with a high capacity rapid stirrer (3000-10,000 r.p.m.) into 980 parts of cold water which had been acidified to pH 5-6 with acetic acid and mixed with 20 g paraffin sulphonate. The emulsion was ready after stirring for 3 to 5 minutes.

The reaction product containing isocyanate groups was obtained in the following way:

3000 parts of a polypropylene glycol having a molecular weight of about 2000, a OH number of 55.5 and an acid number < 0.5 were heated with 535 parts hexamethylene diisocyanate at 110°C for two hours and subsequently at 130-140°C for 1½ hours.

The 40% aqueous copolymer dispersions was obtained by copolymerisation of 60 parts n-butyl acrylate, 30 parts styrene and 10 parts acrylamide in water.

Example 2

A loose wool fabric is treated in a jet washing machine (that is, one in which the washing medium is applied through jets), after washing and without intermediate drying, in a liquor ratio of 1 : 6 at room temperature for about 2 minutes with an aqueous liquor containing, per litre,

27.5 g of a mixture

prepared by stirring into

6 g of the 50% stock emulsion described below first

1.5 g of the 40% aqueous copolymer dispersion described in Example 1 and then

20 ml of water.

Before adding the mixture, the pH value of the liquor had been adjusted to pH 4.5-5.5 by the addition of acetic acid. There is then added to the liquor, per litre,

1.5 g of 30% silica sol,

diluted with 13 ml of water and acidified with acetic acid to pH 5-6,

and the treatment is continued for another 15-20 minutes. Subsequently, there is added to the liquor, per litre,

4.5 g of sodium acetate,

dissolved in 15 ml of water,

and the treatment is again continued for 5-10 minutes. The wool fabric is then centrifuged and dried on a stenter at 90-110°C. The treated wool fabric is characterised by a full handle and excellent felt-free effects, besides very good properties in use, such as resistance to abrasion, creasing and pilling.

The 50% stock emulsion was prepared as described in Example 1, but the isocyanate group-containing reaction product there used was replaced with the reaction product obtained in the following way:

1000 Parts of a branched polypropylene glycol which had been prepared by propoxylation of trimethylolpropane and had a molecular weight of 3750, a OH number of 45 and an acid number < 0.5 was heated with 135 parts hexamethylene diisocyanate at 110°C for two hours and subsequently at 130°C for one hour.

Example 3

A costume fabric of pure wool (pique) is treated in a jet washing machine operating at low gear, in a liquor ratio of 1 : 6 at room temperature for about 10 minutes with an aqueous liquor containing, per litre,

52.2 g of a mixture

prepared by stirring into

10 g of the 50% stock emulsion described in Example 1 first

2.2 g of the 40% copolymer dispersion described in Example 1 and then

40 ml of water.

Before adding the mixture, the pH value of the liquor had been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

1.5 g of 30% silica sol

diluted with 12 ml of water and acidified to pH 5 with acetic acid

and the treatment is continued for another 20 minutes. Subsequently, there is added to the liquor, per litre,

4.5 g of sodium acetate

dissolved in 15 ml of water

and the treatment is continued for another 25 minutes. The wool fabric is then centrifuged and dried at 100°C. There is obtained a wool fabric with an excellent shrink-proof finish and very good properties in use, such as resistance to abrasion and creasing.

Example 4

Pullovers of carded yarn (Shetland wool) are treated in a drum washing machine in a liquor ratio of 1 : 10 at room temperature for about 5-10 minutes with an aqueous liquor containing, per litre,

52 g of a mixture

prepared by stirring into

10 g of the 50% stock emulsion described in Example 2 first

2 g of the 40% copolymer dispersion described in Example 1 and then

40 ml of water.

Before adding the mixture, the pH value had been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

1 g of 30% silica sol

diluted with 10 ml of water and acidified to pH 5-6 with acetic acid

and the treatment is continued for another 20 minutes. Subsequently, the pullovers are centrifuged and dried in a tumbler drier at 80-90°C. An excellent felt-free finish of the pullovers, which is fast to washing, is achieved by the treatment. Moreover, the

knitted fabric has a very good resistance to scraping and pilling.

Example 5

Lambswool pullovers in the grey state are treated in a paddle machine in a liquor ratio of 1 : 30 at room temperature for about 10 minutes with an aqueous liquor containing, per litre,

26 g of a mixture

prepared by stirring into

5 g of the 50% stock emulsion described in Example 1 first

1 g of the 40% copolymer dispersion described in Example 1 and then

20 ml of water.

Before adding the mixture, the pH value of the liquor had been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

0.5 g of 30% silica sol

diluted with 5 ml of water and acidified to pH with acetic acid

and the treatment is continued for another 20 minutes. The pullovers are subsequently dyed by the method conventionally used for knitted articles of wool at boiling temperature from a long bath. After rinsing, centrifuging and drying, the pullovers are shaped and steamed. The pullovers thus treated are characterised by a soft flowing handle and by good resistance to pilling and they have an excellent felt-free finish.

Example 6

Pullovers of pure wool in the grey state are treated in a washing machine in a liquor ratio or 1 : 10 at room temperature for about 10 minutes with an aqueous liquor containing, per litre,

38 g of a mixture

prepared by stirring

30 ml of water into

8 g of the 50% stock emulsion described in Example 2.

Before adding the mixture, the pH value of the liquor had been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

2 g of 15% silica sol

diluted with 20 ml of water and acidified to pH 5-6 with acetic acid

and the treatment is continued for another 15-20 minutes. Subsequently, there is added to the liquor, per litre,

3 g of sodium acetate,

dissolved in 15 ml of water

and the treatment is again continued for 10-15 minutes. After centrifuging and drying in a tumbler drier, the pullovers are dyed by the method conventionally used for knitted articles of wool from a long bath. After another centrifuging and drying at 80-90°C, the pullovers are shaped and steamed. The treated pullovers are characterised by a pleasant handle and very good pilling resistance and they have an excellent felt-free finish.

Example 7

Stockings of pure wool are treated in a drum washing machine in a liquor ratio of 1 : 10 at room temperature for about 10 minutes with an aqueous liquor containing, per litre,

32.5 g of a mixture

prepared by stirring into

6 g of the 50% stock emulsion described in Example 1 first

1.5 g of the copolymer dispersion described in Example 1 and then

25 ml of water.

Before adding the mixture, the pH value had been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

1 g of 15% silica sol

diluted with 10 ml of water and acidified to pH 5-6 with acetic acid.

The treatment is continued for another 15-20 minutes. Subsequently, there is added to the liquor, per litre,

3 g of sodium acetate

dissolved in 20 ml of water

and the treatment is again continued for 15-20 minutes. The stockings are then centrifuged, dried in a tumbler at 80-90°C and subjected to the usual shaping and steaming processes. The stockings thus treated are characterised by outstanding resistance to shrinkage, very good pilling resistance and fastness to scraping.

Example 8

A mixed fabric consisting of 55% of polyacrylonitrile fibre and 45% of wool is treated in a jet washing machine in a liquor ratio of 1 : 6 at room temperature for about 10 minutes with an aqueous liquor containing, per litre,

46 g of a mixture

prepared by stirring into

9 g of the 50% stock emulsion described in Example 2 first

2 g of the 40% copolymer dispersion described in Example 1 and then

35 ml of water.

Before adding the mixture, the pH value of the liquor had been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

1.5 g of 30% silica sol

diluted with 12 ml of water and acidified to pH 5-6 with acetic acid

and the treatment is continued for another 30-40 minutes. Subsequently, there is added to the liquor, per litre,

4.5 g of sodium acetate

dissolved in 15 ml of water.

and the treatment is again continued for 10-

15 minutes. The fabric is then calendered in a continuous calender, subsequently centrifuged and dried on a stenter. The treated fabric is characterised by a full firm handle, very good pilling resistance, fastness to creasing and scraping. It is also dirt-repellent.

Example 9

A mixed fabric consisting of 55% of polyester fibre and 45% of wool is treated in a winch machine in a liquor ratio of 1 : 40 at room temperature with an aqueous liquor containing, per litre,

12.5 g of a mixture

prepared by stirring into

2 g of the 50% stock emulsion described in Example 1 first

0.5 g of the 40% copolymer dispersion described in Example 1 and then

10 ml of water.

Before adding the mixture, the pH value of the liquor had been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

0.4 g of 30% silica sol

diluted with 4 ml of water and acidified to pH 5-6 with acetic acid

and the treatment is continued for another 30-40 minutes. Subsequently, there is added to the liquor, per litre,

1 g of sodium acetate

dissolved in 5 ml of water

and the treatment is again continued for 10-15 minutes. The fabric is then calendered in a continuous calender, subsequently centrifuged and dried on a stenter. The fabric is then treated at 180-185°C for about 30 seconds and finished in the usual way. The treated fabric is characterised by a pleasant full handle, good fastness to wet creasing and good pilling resistance. The wool component also has an excellent felt-free finish.

A fabric with a similar good finish is obtained when the mixed fabric is first thermofixed and then subjected to the treatment with the aqueous treatment liquor described above.

Example 10

Woollen yarn in hanks is treated, after dyeing and rinsing but without intermediate drying, in a liquor ratio of 1 : 40 in a dyeing apparatus for about 10-15 minutes with an aqueous liquor containing, per litre,

10 g of a mixture

prepared by stirring into

2 g of the 50% stock emulsion described in Example 2 first

0.5 g of the 40% copolymer dispersion described in Example 1 and then

7.5 ml of water.

Before adding the mixture, the pH value of the liquor had been adjusted to 4-4.5 by the addition of acetic acid. There is then added to the liquor, per litre,

0.4 g of 30% silica sol

diluted with 4 ml of water and acidified to pH 5-6 with acetic acid

and the treatment is continued for another 15-20 minutes. Subsequently, there is added to the liquor, per litre,

1 g of sodium acetate

dissolved in 5 ml of water

and the treatment is again continued for 10 minutes. The yarn hanks are then centrifuged and dried. The knitted fabrics produced after 1-2 days' storage from the yarn thus treated have an excellent felt-free finish and are characterised by good resistance to scraping, wet creasing and pilling.

Example 11

Undyed wool yarn is treated in a dyeing apparatus in a liquor ratio of 1 : 40 at room temperature for about 10-15 minutes with an aqueous liquor which contains, per litre,

3 g of the 50% stock emulsion described in Example 1 and

0.1 g of an approximately 30% aqueous solution of a fatty alcohol sulphate

and the pH of which has been adjusted to 4.5-5 by the addition of acetic acid. There is then added to the liquor, per litre,

0.4 g of 30% silica sol

diluted with 4 ml of water and acidified to pH 5-6 with acetic acid

and the treatment is continued for another 15-20 minutes. Subsequently, there is added to the liquor, per litre,

1 g of sodium acetate

dissolved in 5 ml of water which has been acidified to pH 5-6 with acetic acid

and the treatment is again continued for about 10 minutes. After the addition of

0.5 g sodium pyrophosphate and

5 g of 35% hydrogen peroxide

per litre of liquor, the treating bath is heated to 50°C within one hour and kept at this temperature for 30 minutes. After draining off the liquor and rinsing, the yarn is dyed in the usual manner from a weakly acidic bath. The knitted fabrics produced from the yarn have an excellent felt-free finish and a full woolly handle, and they are characterised by good resistance to scraping, pilling and wet creasing.

Example 12

A knitted fabric of a textured polyamide fibre is treated in a winch machine in a liquor ratio of 1 : 40 at room temperature for about 15 minutes with an aqueous liquor which contains, per litre,

1.5 g of the 50% stock emulsion described in Example 1 and

0.25 g of a 30% fatty alcohol sulphate solution

and the pH value of which has been adjusted to 4.5-5 by the addition of acetic acid. There

is then added to the liquor, per litre,

0.25 g of 30% silica sol
diluted with 2.5 ml of water and acidified with acetic acid

and the treatment is continued for another 20 minutes. Subsequently, there is added to the liquor, per litre,

1 g of sodium acetate
dissolved in 5 ml of water which has been acidified to pH 5-6 with acetic acid

and the treatment is continued for a further 20 minutes while the bath temperature is raised to 45°C. The knitted fabric is subsequently rinsed, centrifuged and dried. When dyed after 12 hours' storage, the treated knitted fabric is characterised in that it does not exhibit the inconvenient wet stiffness. Furthermore, the finished knitted fabric has a pleasant handle which is fast to washing and dry cleaning, an excellent retention of shape and fastness to creasing.

WHAT WE CLAIM IS:—

1. A process for the finishing of textile material which comprises treating the textile material with an aqueous liquor containing a silica sol and an isocyanate group-carrying reaction product from a compound of molecular weight of from 500 to 6000, containing at least two hydroxyl groups per molecule, with a polyisocyanate of bisulphite addition product thereof.

2. A process according to Claim 1 wherein the textile material is treated with the silica sol-containing aqueous liquor according to the exhaust process.

3. A process according to Claim 1 or 2 wherein the silica sol has an average particle size between 10 and 50 mμ.

4. A process according to any of Claims 1 to 3 wherein the silica sol is added in an amount of from 0.3 to 5 g per litre of treating liquor, the silica sol containing 10-30% by weight of silica.

5. A process according to any of Claims 1 to 4 wherein the isocyanate group-containing reaction product is present in an amount of from 0.2 to 20 g per litre of treating liquor.

6. A process according to Claim 5 wherein the isocyanate group-containing reaction product is present in an amount of from 0.5 to 5 g litre treating liquor.

7. A process according to any of Claims 1 to 6 wherein the treating liquor contains an emulsion stabiliser.

8. A process according to Claim 7 wherein the emulsion stabiliser is a fatty alcohol sulphate or a paraffin sulphonate.

9. A process according to Claim 8 wherein the fatty alcohol sulphate or paraffin sulphonate is present in an amount of from 0.01 to 2 g per litre of treating liquor.

10. A process according to Claim 9 wherein the fatty alcohol sulphate or paraffin sulphonate is present in an amount of from 0.05 to 0.2 g per litre of treating liquor.

11. A process according to Claim 7 wherein the emulsion stabilizer is a polymer or copolymer derived from a vinyl or divinyl monomer.

12. A process according to Claim 11 wherein the polymer or copolymer is present in an amount of from 0.1 to 20 g per litre of treating liquor.

13. A process according to Claim 12 wherein the polymer or copolymer is present in an amount of from 1 to 3 g per litre of treating liquor.

14. A process according to any of Claims 1 to 13 wherein an electrolyte is added to the treating liquor.

15. A process according to Claim 14 wherein the electrolyte is an alkali metal or ammonium salt of an inorganic or organic acid.

16. A process according to Claim 14 or 15 wherein the electrolyte is added in an amount of from 2 to 10 g per litre of treating liquor.

17. A process for the finishing of textile materials comprising agitating the textile material at room temperature in an aqueous liquor in a liquor ratio of from 1 : 6 to 1 : 50, the aqueous liquor containing an isocyanate group-carrying reaction product of a compound of molecular weight of from 500 to 6000 containing at least two hydroxyl groups per molecule with a polyisocyanate, or bisulphate addition product thereof, adding a silica sol to the treating liquor, and allowing the resulting liquor to act on the textile material.

18. A process according to Claim 17 wherein the pH of the liquor prior to the addition of the silica sol is from 4 to 6.

19. A process according to Claim 17 or 18 wherein the treating liquor contains an emulsion stabiliser.

20. A process according to any of Claims 17 to 19 wherein an electrolyte is added after the silica sol-containing liquor has been allowed to act on the textile material.

21. A process according to Claim 1 as hereinbefore described in any of the Examples.

22. Textile materials when treated by a process according to any of Claims 1 to 21.

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